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Source Characterization and Apportionment of the Houston Aerosol by Means of X-Ray Diffraction Techniques

South Dakota School of Mines and Technology
Rapid City

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Although none of the filters analyzed contained aerosol loads sufficiently adequate for optimum XRD analysis, these results compared favorably in many aspects with results obtained by scanning electron microscopy, additional x-ray diffraction work at the Lawrence Berkeley Laboratory, and elemental analyses completed by EPA and other groups.

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by

Briant L. Davis
Institute of Atmospheric Sciences
South Dakota School of Mines and Technology
Rapid City, SD 57701

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EPA Project Officer
Robert K. Stevens

ENVIRONMENTAL SCIENCES RESEARCH LABORATORY
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SOURCE CHARACTERIZATION AND APPORTIONMENT OF THE HOUSTON AEROSOL BY MEANS OF X-RAY DIFFRACTION TECHNIQUES

Briant L. Davis
Senior Scientist, Cloud Physics Laboratory
Institute of Atmospheric Sciences
South Dakota School of Mines and Technology
Rapid City, South Dakota 57701

Eighteen pairs of dichotomous sampler filters (teflon) used in collecting ambient aerosol at Houston, Texas, from 10-18 September 1980, have been studied by x-ray diffractometry. Calcium carbonate from shell fragments constitute the dominating single component of the crustal source material. Lesser amounts of clays, quartz, feldspars, and organic matter are also present. For the ambient filter analyses the coarse fraction was dominated by calcite and quartz, whereas the fine fraction was composed almost exclusively of ammonium sulfate and lead ammonium sulfate. Sea salt aerosol was observed at times in the coarse fraction. Carbon was observed in both coarse and fine fractions.

A source apportionment of the aerosol was completed by least squares minimization. Source composition data were compiled from emission inventories as well as from the analyzed crustal material. Considering average data for the 9-day sampling period, the apportionment revealed that approximately 1/3 of the total aerosol at the receptor was of crustal origin (soil and street dust), fuel oil combustion contributed about 1/2 of the mass (as fine particles) but only 2.0% as coarse particles, transportation contributed about 6% as fine particles and 1% as coarse particles, and ocean aerosol (coarse NaCl) contributed just over 1% to the total aerosol.

Although none of the filters analyzed contained aerosol loads sufficiently adequate for optimum XRD analysis, these results compared favorably in many aspects with results obtained by scanning electron microscopy, additional x-ray diffraction work at the Lawrence Berkeley Laboratory, and elemental analyses completed by EPA and other groups.

SOURCE CHARACTERIZATION AND APPORTIONMENT OF THE HOUSTON AEROSOL BY MEANS OF X-RAY DIFFRACTION TECHNIQUES

1. Introduction

In this paper we report results which are part of a multi-laboratory cooperative effort to develop source apportionment techniques based on several approaches, including elemental analysis, x-ray diffraction analysis (XRD), scanning electron microscopy, and analysis of simulated data. This program is directed by the U.S. Environmental Protection Agency. The analyses were completed on a set of 18 dichotomous filters taken during the period 10-18 September 1980 on the campus of the University of Houston, Houston, Texas. Although the field protocol was not developed specifically with x-ray diffraction included as an analytical tool, recent improvements in XRD quantitative analysis procedures prompted the U.S. Environmental Protection Agency to include this laboratory (IAS) and the Lawrence Berkeley Laboratories (LBL), as participants in the study. For accurate analysis by x-ray diffraction, approximately 200 $\mu g\ cm^{-2}$ aerosol load should be present on the filter. None of the filters satisfied this condition, although sufficient load was present to justify proceeding with the study so that the capabilities of the technique and future needs could be assessed.

2. Experimental Techniques

Actual x-ray diffraction analysis was completed at the Lawrence Berkeley Laboratories using a position-sensitive x-ray detector with computerized instrument control and data acquisition (Thompson et al.[1]). Analyses of the data were completed at the IAS laboratories (as well as at LBL -- to be reported elsewhere) using the data compilation contained by the LBL instrument. Analyses of the soil materials were made using the IAS x-ray diffractometer, which employs copper radiation, and theta compensator, but which requires manual intensity measurements. Component intensities and appropriate physical data were combined in the reference intensity analysis procedure which has been described elsewhere (Davis[2] and Davis and Johnson[3]). Source apportionment calculations included least squares minimization, according to procedures developed by Watson[4] and applied recently by many other investigators (see the review by Gordon[5]).

The analytical and computational aspects of the source apportionment study has resulted in the accumulation of a large volume of tabular data which cannot be presented here, but which may be reviewed in more detail in the report by Davis[6]. In this paper we present only a portion of the total data set with brief comments on the procedure and results.

3. Results

a. Analysis of ambient filters

Although procedures have now been developed to combine x-ray transmission results with x-ray diffraction to obtain a complete amorphous and crystalline component analysis, x-ray transmission data were not available for this set of samples. Therefore, only direct analysis of the

crystalline components could be made. Table I and Table II present the results of the quantitative multi-component analysis of coarse and fine fractions, respectively. The tables also include the analyses for total carbon made by classical combustion procedures at the EPA laboratories. The crystalline components (excluding carbon) sum to 100%. For comparison with other analytical techniques and for source apportionment purposes we have assumed that the carbon material represents the only noncrystalline substance in the sample. This is a reasonable assumption for the Houston study area, although small amounts of fly ash of a siliceous glassy nature, or slag glass, could be present. (These materials have been observed by R. Crutcher for the 9/10/80-0700 samples, using optical microscopy.)

Observation of the magnitude of values in the tables demonstrate that calcite and quartz constitute the major "crustal" components in the coarse fraction, although some ammonium sulfate is also present. It should be noted here that in these analyses a correction for contamination of fine materials in the coarse fraction has been made. Approximately 10% of the fine aerosol may be present in the coarse fraction, which accounts for at least part of the observed "coarse" ammonium sulfate and carbon. These corrections have been made in the data used for the source apportionment calculations described below.

Table II presents the components observed by XRD in the fine fraction of the 18 filter set. Only two components were observed, ammonium sulfate $(NH_4)_2SO_4$ and ammonium lead sulfate $(NH_4)_2 \cdot Pb(SO_4)_2$. One cannot be certain that all of the observed sulfate compounds represent original atmospheric reaction; some portion may represent artifact reaction on the filter. The carbon values in Table II represent measurements by EPA, and may be combined with the components observed by XRD into a total component analysis by simple renormalization.

The EPA steering group for the Houston study has selected two cases, Sep. 10, 0700 and Sep. 13, 0700, along with the average data set for making comparisons between analytical techniques. Table III presents selected elemental ratios for these cases based on analyses completed by x-ray diffraction at the IAS and LBL laboratories, by scanning electron microscopy combined with x-ray energy spectroscopy (SAX) at the State University of New York at Syracuse (SUNY-CESF), and by x-ray fluorescence at EPA. Table IV presents these ratios for the 18 filter set average. Rather large differences, as well as close comparisons, occur in the two individual case days, whereas more general agreement in magnitude occurs for the average set. Most obvious is the low aluminum reduced from the XRD compounds, apparently the result of insensitivity of XRD to the low concentrations of poorly diffracting clay and mica minerals. The low iron content obtained by XRD is also apparent. Fly ash and minor iron oxides are probably the sources in this case. The XRD analysis completed at the LBL was completed by a calibration technique and therefore the differences observed with IAS are not merely the differences in pattern interpretation or intensity measurement but are differences in calibration factors, and reference intensity constants. A comparative study dealing with these difficulties is presently being conducted by the two laboratories.

TABLE I

Quantitative Component Analyses-Coarse Fraction*

Weight Percent
(Variance Error)

	SEP 10		SEP 11		SEP 12		SEP 13	
	0700	1900	0700	1900	0700	1900	0700	1900
Mascagnite $(NH_4)_2SO_4$	8.5 (2.2)	22.6 (4.9)	24.8 (5.2)	41.7 (6.8)	34.0 (6.3)	42.4 (6.8)	23.2 (5.0)	24.6 (5.2)
Quartz SiO_2	43.2 (3.3)	38.1 (3.7)	35.1 (3.7)	27.2 (3.9)	32.0 (4.0)	21.2 (3.2)	38.0 (3.8)	20.8 (2.5)
Calcite $CaCO_3$	48.3 (3.1)	28.1 (2.5)	21.1 (2.1)	25.5 (3.3)	34.0 (3.7)	26.6 (3.4)	38.8 (3.4)	54.6 (4.2)
Dolomite $(Ca,Mg)CO_3$	--	11.3 (1.6)	19.0 (2.6)	5.6 (1.8)	--	9.8 (1.7)	--	--
Halite NaCℓ	--	--	--	--	--	--	--	--
Carbon (TOTAL)	10.2 (1.4)	15.5 (2.4)	12.5 (2.2)	16.8 (3.8)	14.2 (3.0)	17.0 (2.9)	10.2 (1.5)	20.8 (5.4)
$\tilde{M}_B$ ($\mu gm\ cm^{-2}$)	80	52.9	52.9	34.9	40.9	42.0	76.9	22.9
$\tilde{M}_V^B$ ($\mu g\ m^{-3}$)	38.4	22.6	23.2	13.7	17.6	18.2	34.3	9.6

Weight Percent
(Variance Error)

	SEP 14		SEP 15		SEP 16		SEP 17		SEP 18	
	0700	1900	0700	1900	0700	1900	0700	1900	0700	1900
Mascagnite $(NH_4)_2SO_4$*	12.8 (3.1)	19.6 (4.4)	--	10.3 (2.6)	6.8 (1.8)	--	--	--	--	--
Quartz SiO_2	27.9 (2.8)	26.3 (3.0)	42.1 (3.2)	36.9 (3.1)	34.9 (3.0)	8.6 (1.0)	35.0 (3.0)	19.2 (2.0)	23.1 (2.3)	22.4 (2.3)
Calcite $CaCO_3$	59.3 (3.2)	17.8 (1.7)	57.9 (3.2)	48.4 (3.0)	58.3 (3.0)	48.9 (2.4)	65.0 (3.0)	53.2 (2.3)	46.8 (2.4)	60.5 (2.6)
Dolomite $(Ca,Mg)CO_3$	--	36.3 (3.9)	--	4.4 (0.6)	--	18.4 (2.2)	--	9.4 (1.2)	6.9 (0.9)	17.2 (2.1)
Halite NaCℓ	--	--	--	--	--	24.0 (2.1)	--	18.2 (1.7)	23.2 (2.0)	--
Carbon (TOTAL)	11.9 (2.5)	16.6 (3.6)	10.8 (1.8)	12.8 (1.7)	10.3 (1.5)	20.2 (4.4)	9.2 (1.7)	14.2 (2.1)	13.4 (1.9)	9.7 (1.2)
$\tilde{M}_B$ ($\mu gm\ cm^{-2}$)	48.0	30.0	60.0	64.9	72.9	24.0	60.9	48.9	52.0	86.0
$\tilde{M}_V^B$ ($\mu g\ m^{-3}$)	21.2	14.5	29.6	29.8	34.8	11.9	30.3	24.7	26.8	45.3

*Analysis of all components (except carbon) completed by x-ray diffraction (RIM). Sum of all components except carbon equals 100%. Carbon analyses provided by EPA (performed by classical combustion procedures) are given as percent of total mass.

TABLE II

Quantitative Component Analysis - Fine Fraction*

		Weight Percent (Variance Error)					
		$(NH_4)_2SO_4$		$(NH_4)_2Pb(SO_4)_2$		Carbon	$\breve{M}_B$ ($\mu g\ cm^{-2}$)
SEP 10	0700	100		--		18.3	92.0
	1900	85.5	(13.2)	14.5	(13.2)	18.1	100.9
SEP 11	0700	89.3	(7.7)	10.7	(7.7)	15.6	92.0
	1900	99.5	(0.4)	0.5	(0.4)	11.5	88.0
SEP 12	0700	80.9	(13.4)	19.1	(13.4)	11.2	76.9
	1900	100		--		14.2	74.9
SEP 13	0700	83.5	(10.3)	16.5	(10.3)	14.9	122.0
	1900	78.8	(14.9)	21.2	(14.9)	19.1	46.9
SEP 14	0700	98.0	(1.5)	2.0	(1.5)	16.0	78.9
	1900	100		--		27.6	28.9
SEP 15	0700	100		--		27.0	48.0
	1900	99.3	(0.5)	0.7	(0.5)	20.9	89.0
SEP 16	0700	94.8	(3.8)	5.2	(3.8)	20.0	78.9
	1900	94.8	(11.2)	5.2	(11.2)	35.8	18.0
SEP 17	0700	90.4	(6.9)	9.6	(6.9)	25.5	44.9
	1900	100		--		24.4	30.0
SEP 18	0700	100		--		30.4	22.9
	1900	100		--		23.0	20.9

*Analysis of all components (except carbon) completed by x-ray diffraction (RIM). Sum of all components except carbon equals 100%. Carbon analyses provided by EPA (performed by classical combustion procedures) given as percent of total mass.

TABLE III

Selected Elemental Ratios Calculated for Two Case Days of the Houston Study, A, 9/10-0700; B, 9/13-0700, Coarse Fraction

METHOD	Ca/Si		Al/Si		Fe/Si		Mg/Si		S/Si	
	A	B	A	B	A	B	A	B	A	B
SAX	1.06	2.02	0.13	0.25	0.06	0.36	0.03	0.01	0.10	0.15
XRD (IAS)	0.92	0.84	0	0	0.02	0.02	0	0.003	0.10	0.32
XRD (LBL)	0.97	2.12	0	0	0.12	0.56	0.01	0.15	0.38	0.94
XRF	0.78	0.84	0.35	0.37	0.27	0.24	--	--	0.22	0.14

TABLE IV

Selected Elemental Ratios, averaged for the Houston 18-filter set, coarse fraction

METHOD	Ca/Si	Al/Si	Fe/Si	Mg/Si	S/Si
SAX	1.77	0.19	0.30	0.02	0.10
XRD (IAS)	1.52	0.04	0.04	0.08	0.19
XRD (LBL)	2.18	0.02	0.03	0.06	0.56
XRF	0.93	0.37	0.20	--	0.13

b. Soil and street analyses

Table V presents the analyses of the three soil and street dust samples collected in the vicinity of the receptor site at the University of Houston. The major component observed here, which is not observed in the ambient filters, is the clay montmorillonite. This is not surprising when considering the structural characteristics of this material, since it is well known that individual montmorillonite crystallites tend to swell and agglomerate in the presence of moisture and resist saltation into the atmosphere. The high calcite component in the Shell road and Wheeler road analyses account for the major amounts of this component observed in the ambient analyses. Also included in Table V are element concentrations in weight percent obtained by stoichiometric reduction of the known compounds in the multi-component analysis.

TABLE V

Receptor Site Soil and Street Dust Analyses
(Compounds and Reduction to Elements)

Component	Weight Percent		
	Shell Rd. (UHS-1)	Library (UHS-2)	Wheeler Rd. (UHS-3)
Kaolinite	0	0.8	2.9
Montmorillonite	13.6	34.4	19.0
Quartz	33.8	41.5	37.7
Microcline	0.9	2.3	3.0
Andesine	0	1.7	1.2
Calcite	50.5	18.8	33.9
Dolomite	0	0.7	2.4
Organic Matter	1.2	0	0
ELEMENT:			
Si	20.0	30.6	24.9
Al	1.7	4.7	3.2
Na	0.3	0.8	0.5
K	0.1	0.6	0.6
Ca	19.5	7.7	13.7
Fe	0.7	1.0	0.8
Ni	0.5	0.2	0.4
Mg	0.3	0.7	0.6
Mn	0.4	0.2	0.3
$C(CO_3)$	6.1	2.3	4.4
C(Free)	1.2	0	0
H_2O	0.8	2.1	1.5

4. Source Apportionment Computations

On-site sampling of the large number of point sources in the vicinity of the receptor was not available for the apportionment analysis. Because of the inability of the x-ray diffraction analyses to quantitatively evaluate minor components at such light filter loadings, only a few major source categories were included in the apportionment study. These are transportation (F_1), fuel oil combustion (F_2), soil (F_3), streets (F_4), and ocean aerosol (F_5). Table VI presents the source matrix used in the apportionment calculations. These source compositions were obtained from various published inventory source data including a NEDS[7] compilation for Harris County, Texas. The composition for the ocean aerosol was taken from Deer et al.[8], and Holmes[9], and represents the nominal composition for sea water. The following factors and assumptions are noted in the development of the source matrix.

1. Twenty percent of the gaseous hydrocarbon emissions were assumed to be converted to carbon bearing particulate matter;

2. The weight ratio of lead to carbonaceous matter (particulates) of 0.166 was taken as an average of one value calculated directly from carbon and lead emission data and one value quoted in the literature (Dzubay et al.[10]); and

TABLE VI

Source Compositions

Weight Percent (Variance Error)

Component	F_1 Transportation	F_2 Combustion	F_3 Soil	F_4 Streets	F_5 Ocean Aerosol
Quartz SiO_2	0	0	68.6 (4.7)	45.5 (4.7)	0
Calcite $CaCO_3$	0	0	30.4 (2.5)	52.6 (4.7)	0.7 (1.1)
Dolomite $(Ca,Mg)CO_3$	0	0	1.0 (0.1)	1.4 (0.2)	0
Halite NaCl	0	0	0	0	90.4 (9.0)
PbO	12.7 (4.1)	0	0	0	0
$SO_4^=$	6.4 (6.2)	72.3 (8.7)	0	0	8.9 (8.9)
Carbon	80.9 (2.2)	27.8 (8.7)	0	0.5 (5.6)	0

3. Emission factors of 0.66 gram lead and 13.91 gram carbon per liter of gasoline consumed are obtained from the lead and carbon emission data.

The lead sulphates were recalculated to lead oxide for both the ambient and source composition, although in actuality the lead may occur as PbBrCl, or $PbBr_2$, etc. in the atmosphere. The nature of the lead species is irrelevant in the source apportionment calculations providing one is consistent in both source and filter matrices.

The error quantities used in the least source apportionment were obtained from the variance error calculations (Davis[11]) for both the ambient and soil/street dust analyses, and were estimated for the source inventory data by the several methods of computation or of the differing values described in the literature.

Table VII presents the contribution factors (in $\mu g\ m^{-3}$) determined for the two case days and for the 36-filter weighted mean. All data represent the fourth iteration in the effective variance least squares procedure. Table VIII presents the observed component compositions (as used in the source apportionment matrix) and those values calculated from the contribution factors obtained at the fourth iteration.

Contributions from soils (F_3) and streets (F_4) were computed separately, as implied in Table VI; similarities in source compositions resulted in negative values for F_3 in over half of the 18 cases run. This colinearity problem was solved by adding the F_3 and F_4 factors into a single "crustal" source, the resultant of which was a positive number in all cases.

The most dominant feature of these results is the crustal contribution to the coarse fraction and the significant fuel oil combustion contribution to the fine fraction. The analyses for the ambient filters for fine and coarse fractions were combined when completing the source apportionment calculations and then reapportioned into coarse and fine components on the basis of the sulfate for combustion and carbon for transportation. This was necessitated by the fact that only two components were present in the fine fraction of the filter analyses which would not readily lend itself to source apportionment computations. From the average data we observe that approximately 38% of the aerosol observed at the receptor is of crustal origin, whereas 55%, the dominant species overall, derives from combustion sources. A small amount of sea salt is present in a number of samples collected at the end of the sampling period. If one examines the trend of the composition factors from beginning to end of the Houston sampling period, one notices two major segments of aerosol transport which are related to air mass and wind conditions. This is displayed in Figure 1 where wind velocity and resultant 6 hour average wind direction parameter are plotted against sampling time. Also superimposed on these data are the 12-hour values of the ratio F_4/F_2, or the ratio of street contributions to combustion contributions. Also plotted is the extinction coefficient measured during the study as reported by Dzubay et al.[12]. The hourly data set was provided by Dr. C. W. Lewis, EPA/RTP. The change in air mass correlates with beginning of an upward trend in F_4/F_2 as shown by a change in

TABLE VII

Contribution Factors for September 10 (0700), September 13 (0700), and for the 9-day (36-filter) Sampling Period

Date/ Time	Units	F_1 Transportation		F_2 Combustion		F_3+F_4 Crustal	F_5 Ocean	Total
		Coarse	Fine	Coarse	Fine	Coarse	Coarse	
	%	0.05	0.5	1.2	53.7	44.5	0	100.0
9/10/80	$\mu g\ cm^{-2}$	0.07	1.0	1.6	71.4	59.1	0	133.2
0700	$\mu g\ m^{-3}$	0.05	0.5	1.1	50.8	42.1	0	94.6
	%	1.1	7.2	3.5	57.4	31.6	<0.01	100.8
9/13/80	$\mu g\ cm^{-2}$	1.1	7.2	3.5	57.3	31.5	<0.01	100.6
0700	$\mu g\ m^{-3}$	1.2	7.7	3.8	61.8	34.1	<0.01	108.6
Mean	%	0.8	5.6	2.0	55.4	38.3	1.3	103.4
36	$\mu g\ cm^{-2}$	1.0	6.5	2.4	64.8	44.7	1.5	120.9
Filters	$\mu g\ m^{-3}$	0.5	3.6	1.3	35.2	24.3	0.8	65.7

TABLE VIII

Observed and Calculated Component Concentrations for Case-day Filters and 36-filter Mean

$\mu g\ m^{-3}$

Component	9/10/80-0700		9/13/80-0700		Mean 36-filters	
	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
Quartz	19.6	19.6	16.6	16.6	8.5	9.3
Calcite	21.9	21.8	17.0	16.9	13.4	14.4
Dolomite	0	0.6	0	0.5	2.4	0.4
Halite	0	0	0	0.004	1.3	0.7
PbO	0	0.06	5.6	1.1	1.1	0.5
$SO_4^=$	37.4	37.6	52.1	48.0	24.7	26.7
Carbon	15.8	15.1	16.5	25.6	12.0	13.2
Totals	94.7	94.8	107.8	108.7	63.4	65.2

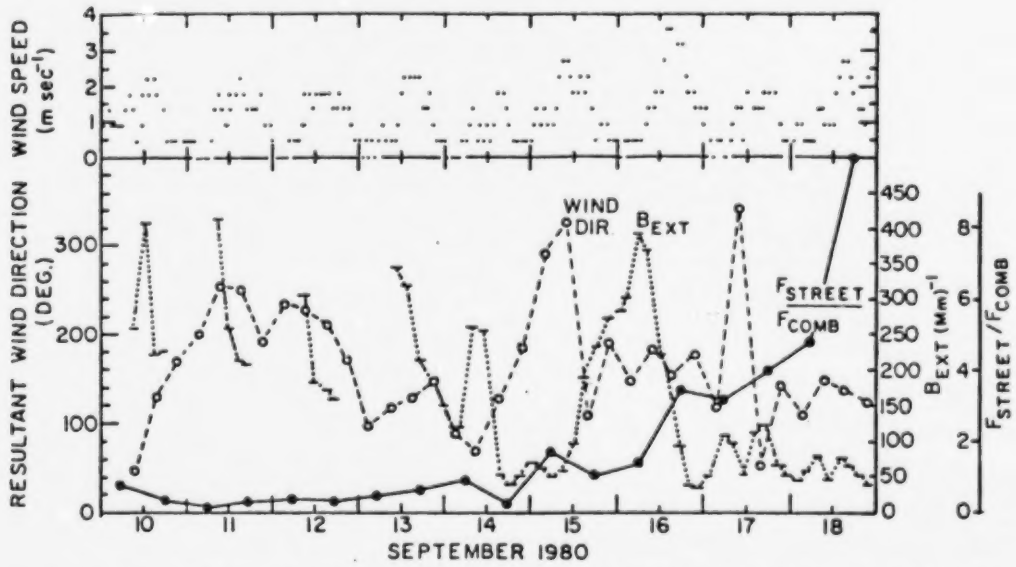

Figure 1. Variation of wind speed, resultant wind direction, extinction coefficient, and source contribution ratio F_{STREET}/F_{COMB} through the sampling period Sep 10-18, 1980. Site is University of Houston campus. B_{EXT} values earlier than 0900, Sep 14, contain ±15% normalization uncertainty.

the wind speed maximum during the afternoons, and shift to a more southeasterly wind direction. The increase in wind velocity signals the dilution of the combustion aerosol trapped over the city and creates a corresponding traffic-induced increase in fugitive dust sources at the sampling site.

5. Conclusions

X-ray diffraction analysis has been applied to the Houston aerosol for compound characterization and source apportionment based on broad source categories. Results show that the major contribution to the aerosol in the coarse fraction is of crustal origin, and the major contribution in the fine fraction from fuel oil combustion. Even though the ambient filters were not loaded with sufficient aerosol mass to achieve optimum XRD sensitivity these results are in reasonable agreement with those of companion studies completed by other techniques. A good correlation of source contribution trends with observed meteorological conditions may be seen over the 9-day sampling interval at the University of Houston sampling site.

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